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Docket No.: P0806.70004US00
(PATENT)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Robert J. Dobbs
Serial No.: 10/797343
Confirmation No.: 4289
Filed: March 10, 2004
For: MULTI-CARBIDE MATERIAL MANUFACTURE AND USE
Examiner: A. T. Raetzsch
Art Unit: 1754

CERTIFICATE OF FACSIMILE TRANSMISSION 37 C.F.R. §1.8(a)
I hereby certify that this paper (along with any paper referred to as being attached or enclosed) is being transmitted by facsimile to the Patent and Trademark Office, facsimile no. (571) 273-8500, on the date shown below.

Dated: 9/12/06

Signature

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Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

DECLARATION OF ROBERT J. DOBBS UNDER 37 C.F.R. §1.132

I, Robert J. Dobbs, do hereby declare that:

1. I have over 30 years experience in new materials technologies including inventing, manufacturing, marketing, and commercializing new materials. I have 25 years experience in fine particle technology including process technologies for making fine particles such as milling. I worked for 21 years at GTE Products Corporation (Stamford, CT) where I worked on particle processing and milling issues including studying mill wear and developing milling media materials.

I am a founder and currently the Chief Technical Officer of Primet Precision Materials, Inc., who has rights to the above-referenced patent application. I have an ownership interest in Primet Precision Materials. Primet Precision Materials is a materials technology company focused on developing nanomaterials (e.g., materials having a particle size of less than 100 nm) using technology described and claimed in the above-referenced patent application.

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I have a B.S. from the University of Pennsylvania in Materials Science.

2. I am the sole inventor of the above-referenced patent application. I have reviewed the above-referenced patent application, the amendment filed on August 22, 2006 including the amended claims, and the Office Action mailed February 22, 2006, as well as the references cited in the Office Action including U.S. Patent Nos. 3,737,289 and 3,840,367 (the Rudy patents) and U.S. Patent Publication No. 2002/0047058 (Verhoff).

3. One aspect of the invention claimed in the above-referenced application is the use of grinding media formed of multi-carbide material for milling product in a media mill. As described further below, using such grinding media would not have been obvious to one of ordinary skill in the art before the invention that led to the above-referenced application. Moreover, using such grinding media solves several long standing problems in the milling industry and enables unexpectedly good results, as described further below. Also, my colleagues and I at Primet Precision Materials typically encounter significant initial skepticism from experts in the milling and/or small particle industry when we talk about this discovery and the results that we can achieve, as described further below.

I co-founded Primet Precision Materials based on the discovery that grinding media formed of multi-carbide material could be used to mill product in a media mill to form advanced particle compositions including nanoparticle compositions (e.g., average particle size of less than 100 nm) and having little or no contamination (e.g., less than 800 ppm). In my experience, I had not encountered any milling process that was capable of producing such compositions prior to my discovery that led to the above-referenced application. For 12 years, I investigated numerous different materials (on the order of 40-50) as grinding media for milling product in a media mill before discovering that multi-carbide grinding media could be used to form small particle compositions having small average particle sizes and low contamination levels. This discovery was entirely unexpected to me and, I would expect, to those of ordinary skill in the milling field.

4. It is my opinion that the Rudy patents fail to teach or suggest any type of grinding media suitable for milling product in a media mill or any type of method for milling product in a milling

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process. The Rudy patents disclose multi-carbide metal cutting tools which are not suitably shaped for milling product in a media mill and, thus, are structurally different than the grinding media used in media milling processes. Though the Rudy '367 patent does mention using materials for certain applications (e.g., wear-resistant linings in mills) for "milling and drilling" in the mining industry, it is my view that this does not relate in any way to using those materials as grinding media for milling a product in a media mill.

5. It is my opinion that one of ordinary skill in the art would not have been motivated to use the multi-carbide material described in the Rudy patents as grinding media for milling product in a media mill prior to the invention that led to the above-referenced application. Specifically, one would not have been motivated to use the multi-carbide material described in the Rudy patents as the grinding media in Verhoff.

At the time of the filing date of the present application, conventional wisdom in the milling field was that materials having the properties (e.g., superhard, extremely high wear resistance) of multi-carbide materials would not have been suitable grinding media for milling product in a media mill. If used to mill product in a media mill, conventional wisdom taught that grinding media having such properties would be detrimental to the milling process. For example, such superhard grinding media would be expected to damage the milling equipment and/or fracture one another. In my experience working with hard grinding media prior to my discovery that led to the above-referenced application, I have found that to be the case. Therefore, it is my view, based on the properties of multi-carbide material, that one of ordinary skill in the art would not have been motivated to use multi-carbide material as grinding media.

6. My invention solves long standing problems associated with milling technology that have severely limited the use of milling processes for production of many types of very small particle compositions. For example, milling with multi-carbide grinding media can eliminate the problem of contaminating the milled product during processing (e.g., due to wearing of grinding media). As discussed herein and in the above-referenced patent application, multi-carbide material grinding media can be used to form milled compositions having little or no contamination. Also, milling with multi-carbide material grinding media can remove particle size reduction limitations typically

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associated with milling. The multi-carbide material grinding media enables production of very small particle compositions from a wide variety of materials having average sizes that were previously unobtainable with milling. At the time the invention that led to the above-referenced application was made, experts in the milling field generally believed that milling processes were not capable of producing small particle compositions having small average particle sizes (e.g., less than 100 nm) and low contamination levels (e.g., less than 800 ppm).

7. I, and my colleagues at Primet Precision Materials, typically encounter significant initial skepticism from experts in the milling and/or small particle industry when we talk about this discovery and surprise (or, even, disbelief) when we provide evidence of it. For example, one author of a book on milling technology originally told one of my colleagues that primary crystallites of titania cannot be sized reduced and, after witnessing our results which showed such reduction, said that he was very impressed and had been wrong with his original contention. At a recent industry conference, where Primet Precision Materials presented results showing nanoparticle compositions obtained using multi-carbide material as grinding media, someone in the audience expressed that the results were "like a miracle" and asked how they were achieved. On a recent trip visiting customers of Primet Precision Materials in Japan, after presenting our results related to production of titania nanoparticles using multi-carbide grinding media, I was congratulated by a high level manager because his company spent years trying to improve media milling processes and concluded that making titania nanoparticles by milling was simply not possible. I am aware of many other similar examples.

8. I, and others at my direction, have demonstrated unexpectedly good results when using multi-carbide grinding media in a milling process in connection with the production of a wide variety of different material compositions. The unexpected results include production of compositions having a very small particle size (e.g., an average particle size of less than 100 nm and even much lower) and a low contamination level (e.g., less than 800 ppm and even much lower). In all my experience, I am not aware of any milling process being capable of producing such particle sizes and contamination levels.

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The results have been obtained with a wide variety of different product materials including oxides, carbides, carbonates, glass, metals, organics, phosphates, and semiconductors. Specific materials have included aluminum oxide, barium oxides (e.g., barium titanium oxide, barium zirconium oxide, barium ruthenium oxide), cerium oxide, copper oxide, indium tin oxide, lead zirconium titanium oxide, titanium dioxide (e.g., rutile, anatase, brookite), tungsten oxide, silicon carbide, calcium carbonate, alkali zinc phosphate, ordered intermetallics (nickel bismuth, platinum bismuth, platinum lead), carbon, coal, pigments, lithium metal phosphates, gallium nitride (pure or doped), diamond, and silicon.

With many materials, average particle sizes of less than 50 nm have been achieved. In some cases, average particle sizes of less than 20 nm and, even less than 10 nm and 5 nm, have been achieved. In some cases, the particles are so small that they become difficult to analyze using available instrumentation.

Exhibits A-E are microscopy images of samples of different material compositions produced in milling processes that use grinding media formed of multi-carbide materials.

9. I have also compared the performance of multi-carbide grinding media material with a commercially-available yttria-stabilized zirconia grinding media (Zirmil[®]) available from Saint-Gobain ZirPro; www.zirpro.saint-gobain.com). I selected the Zirmil[®] grinding media for comparison because it is recognized in the field as a very high performing grinding media.

The experiments used a laboratory scale media mill (LabStarTM available from Netzsch; www.netzschusa.com) having a chamber volume of approximately 0.6 litre. The mill was equipped with a peristaltic pump to circulate the particle slurry and a heat exchanger to keep temperature constant.

Experiments were conducted with (a) a yttria-stabilized zirconia grinding media; and (b) a tungsten-titanium carbide-based multi-carbide grinding media. The same procedures were followed for both types of grinding media. The chamber fill in the mill was 85% by volume. 100 grams of rutile pigmentary titania with an average particle size of 325 nm was used as the feed material. The titania was dispersed in 400 grams of water to achieve a 20% solids loading. The slurry pH was adjusted to 10-10.5 and kept constant throughout the experiment using AMP. 0.5 grams of a dispersant was used to reduce the viscosity. The pump speed was set at 300 rpm and the agitator

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speed was set at 1800 rpm. Energy consumption (kj/kg) was calculated from the output power which was measured during milling. At various stages of the milling process, particles were collected and analyzed to measure particle size using a DT1200 acoustic analyzer (available from Dispersion Technologies Inc.; www.dispersion.com).

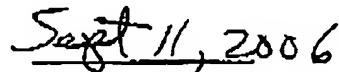
Exhibit F is a graph of energy consumption versus average particle size (D_{50}) for the two types of grinding media. As can be seen from the graph, significantly improved performance was obtained using the multi-carbide grinding media. The improvements include significantly better efficiencies using the multi-carbide grinding material (e.g., 10 times more efficient at 50 nm) and the ability to obtain significantly smaller particle sizes (10 nm) with the multi-carbide grinding media. We did not find it possible to produce titania having an average particle size of less than 50 nm with the Zirmil^(R) grinding media.

At 50 nm particle size, the grinding media were weighed and compared to their initial weight. The Zirmil^(R) grinding media had lost about 4% weight which could be attributed to wearing of the grinding media during milling. That weight percentage led to contamination levels significantly greater than 800 ppm. The multi-carbide material grinding media weight change was not measurable and led to contamination levels of less than 800 ppm.

10. I declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.



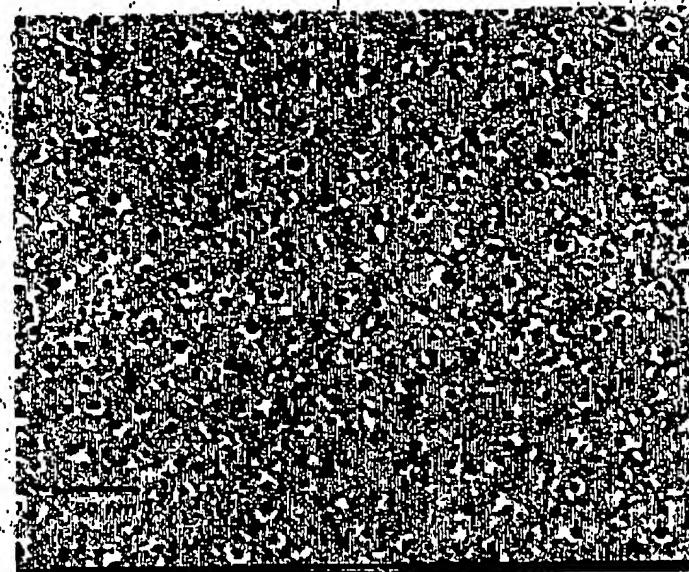
Robert J. Dabbs



Date

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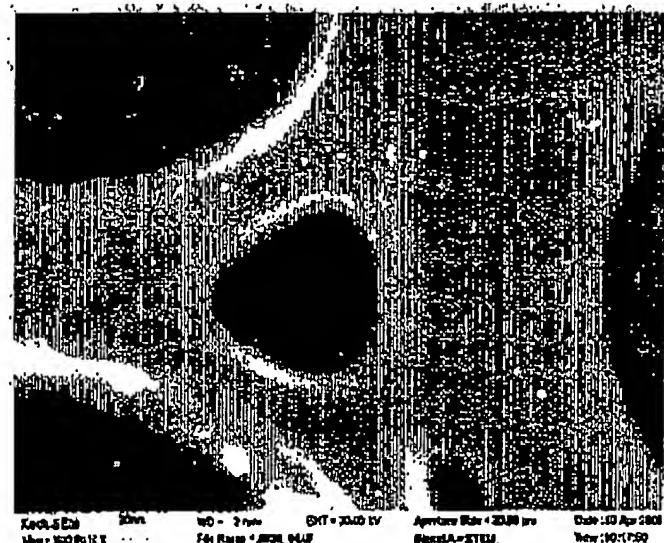
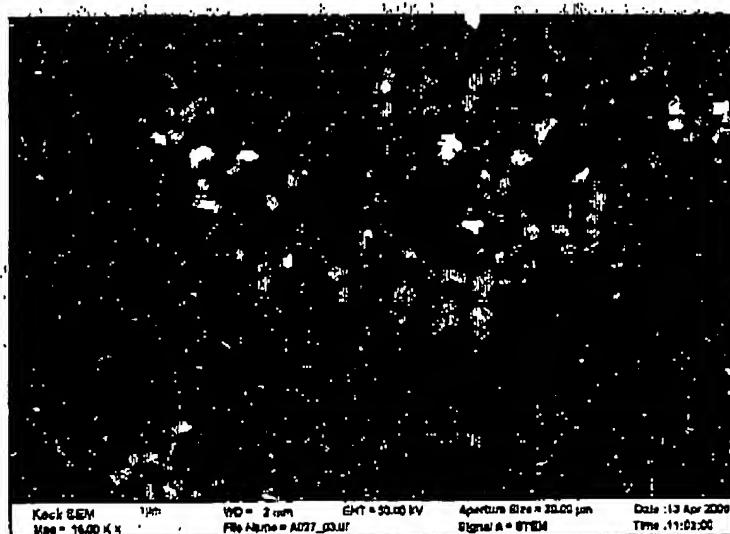
EXHIBIT A

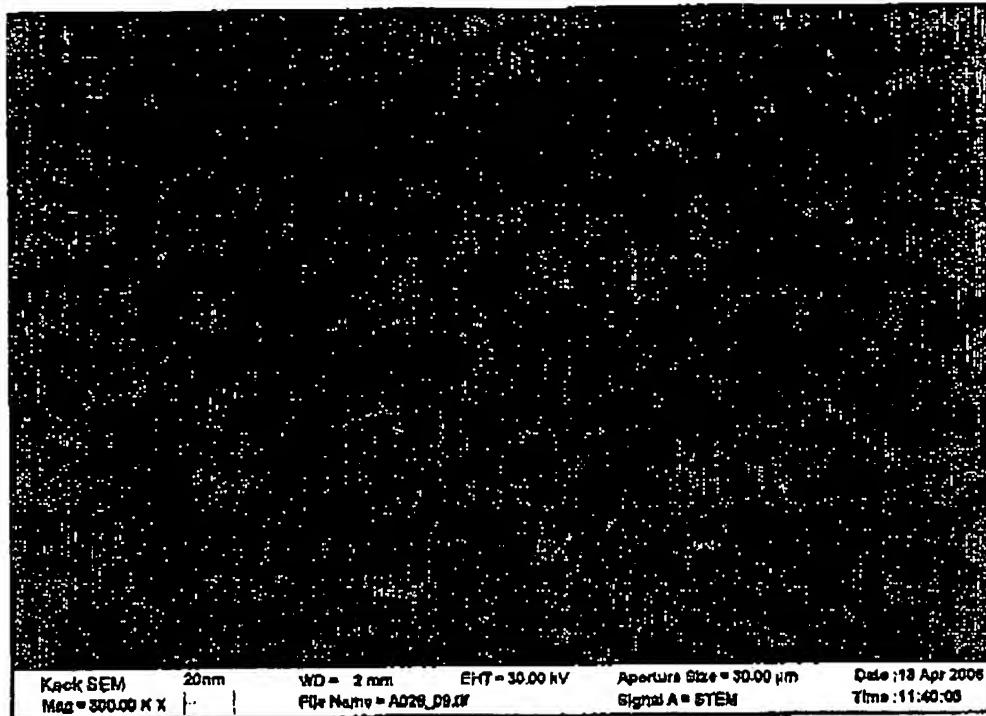


10 nm TiO₂ Spheres

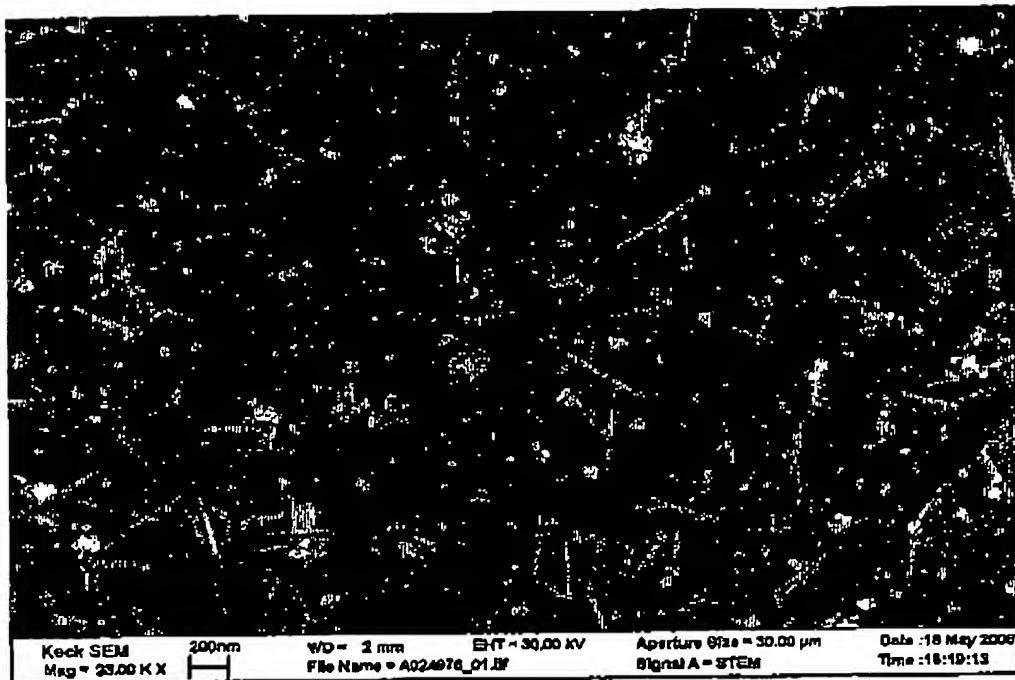


30 nm TiO₂ Platelets

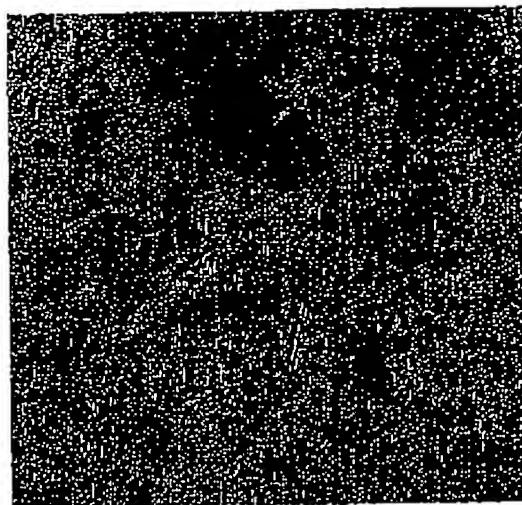
3 nm Ti_2O_3 20 nm TiO



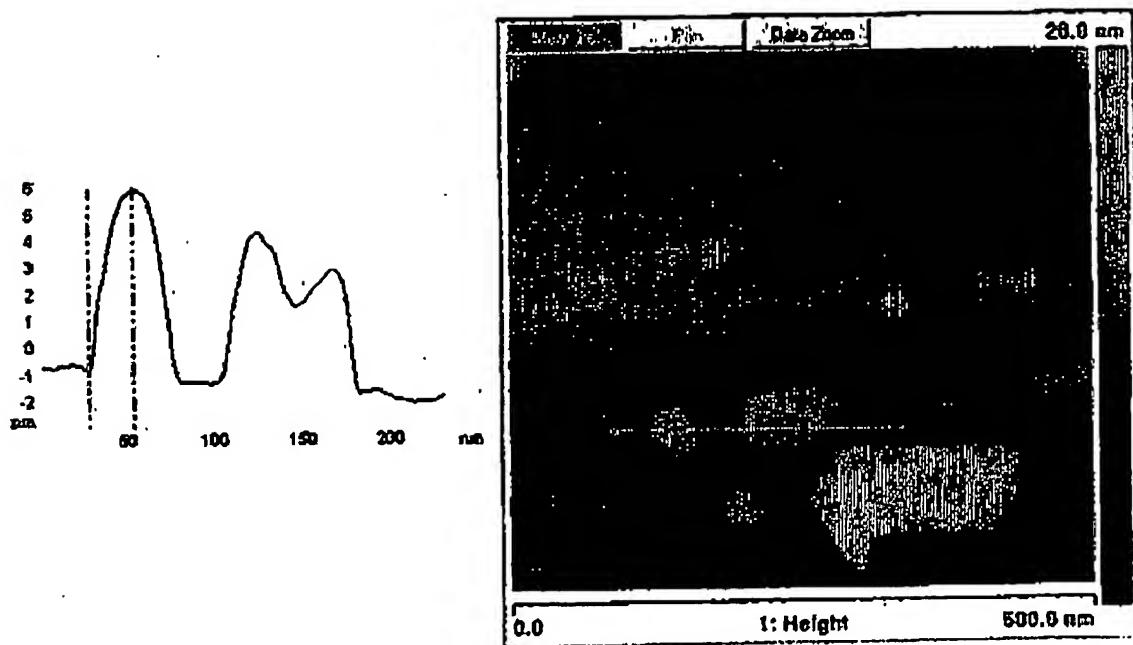
- Magenta Polymer Pigment
- Average particle size is 2 nm
- Size Bar on Photo is 5 nm



- ITO Particles
- Rods
 - >200 nm Length
 - <10 nm Diameter
 - Spheres
 - 10 to 50nm Diameter



FESEM image of Si nanoparticles



AFM image of Si nanoparticles and line scan showing that particles are about 50 nm in length and 6 nm in thickness

Energy Efficiency Zimil™ vs Multicarbide

